# Orthodontic material applications over the past century: Evolution of research methods to address clinical queries

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Abstract: The advances in the field of materials as they relate to orthodontics can be divided into the actual evolution of materials applied to daily practice and the changes in research methods to study the performance and the biologic properties of the materials. Although it is evident that new materials have saturated the market during the past century, the basic concepts of attaching one appliance to the enamel to use as a grip and inserting wires into that to control the spatial orientation of a tooth are identical to the original concepts. In contrast to that, the numbers of treatises about those subjects and the complexity of instrumentation and analytic tools used in published research have advanced tremendously and at a frenetic pace. This highly specialized pattern of research may effectively raise boundaries across research areas, since the complexity of the issues allows researchers to comprehend the content of journal articles in a narrow spectrum of disciplines. The purposes of this article were to review the advances in the research methods for investigating the various properties of orthodontic materials and to assist the reader in navigating this topic. A synopsis of the materials is also provided, listing future applications that already exist at the experimental stage or are yet unavailable but with the relevant technology already presented in broader scientific disciplines.

DOI: https://doi.org/10.1016/j.ajodo.2015.03.007



Originally published at:
Eliades, Theodore (2015). Orthodontic material applications over the past century: Evolution of research methods to address clinical queries. American Journal of Orthodontics and Dentofacial Orthopedics, 147(5 Suppl):224-231.
DOI: https://doi.org/10.1016/j.ajodo.2015.03.007

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# Orthodontic material applications over the past century: evolution of research methods to address clinical queries

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**ABSTRACT**

The advances in the field of materials as it relates to Orthodontics can be divided in the actual evolution of materials applied to daily practice; and the change of research methods to study the performance of the materials as well as their biological properties. Although it is evident that new materials have saturated the market during the past century, the basic concept of attaching one appliance to the enamel to use it as a grip and inserting wires into that to control tooth spatial orientation is identical to the original concept. In contrast to that, the level of treatise of subjects and the complexity of instrumentation and analytical tools employed in published research have advanced to a tremendously high extent and at a delirious pace. This highly specialized pattern of research may effectively raise boundaries across research areas, in the sense that the complexity of issues allows researchers to comprehend the content of journal articles in a narrow spectrum of disciplines. The purpose of this text is to review the advances occurring in the field of research methods of investigating various properties of orthodontic applications of materials, to assist the reader in navigating into this topic; a synopsis of the future of materials is also provided listing future applications, which already exist at experimental stage or are yet unavailable but with the relevant technology already presented in broader scientific disciplines.

## INTRODUCTION

The application of dental materials science in orthodontics coincides with the use of gold wire alloys by E. Angle; the father of the specialty might have not imagined the impact materials would have had in current orthodontic practice. As the field progressed and grew to receive the dimensions of a specialty, the incorporation of principles of materials and mechanics of materials, which are typically taught in the first year of an undergraduate engineering curriculum, were introduced in postgraduate orthodontic curricula along with accompanying elements of materials.

In the United States particularly, the disciplines orthodontic mechanics and materials science, received further emphasis, possibly because of the requirement for a previously earned degree prior to entering dental school. This prerequisite allowed for the cultivation and growth of the materials research, since dental graduates entering orthodontic programs, were often equipped with a bachelor's level of formal training in natural or engineering sciences, thus bringing a new perspective to traditional and empirically-taught concepts of the topic.[1]

The advances in the field of materials as it relates to Orthodontics can be divided in the actual evolution of materials applied to daily practice; and the change of research methods to study the performance of the materials as well as their biological properties.

With respect to the first section, although it is profoundly evident that new materials have saturated the market during the time period examined, the basic concept of attaching one appliance to the enamel to use it as a grip and inserting wires into that to control tooth spatial orientation is identical to the original concept. Of course there have been different modes of attaching this 'handle' to the tooth structure along with a large variation of bracket materials and designs (lingual, self-ligating), and more alloy selection option are available, however the foregoing changes are within the path of the original concept.

In as much, aligners have revolutionized the conventional appliance configuration and constitute a new means of tooth movement, nonetheless, they too represent a new version of removable appliance tooth movement pattern which was popular in the middle of the previous century. Collectively, the orthodontist practicing in the E. Angle era could adapt with only a few hours briefing seminar without much of difficulty to the conditions of the profession existing 100 years later.

In contrast to that, the level of treatise of subjects and the complexity of instrumentation and analytical tools employed in published research have advanced to a tremendously high extent and at a delirious pace. For example, the engineering approaches in materials science and mechanics, or the methods utilized to study the biological mechanisms of tooth movement, as well as the data analysis attributes of clinical trials, require a strong background in the respective sciences involved, which makes it almost unattainable even for the contemporary orthodontist to follow the developments across fields. This highly specialized pattern of research may effectively raise boundaries across research areas, in the sense that the complexity of issues allows researchers to comprehend the content of journal articles in a narrow spectrum of disciplines. This has been highlighted by investigations on the characteristics of orthodontic publications, which showed a significantly higher frequency of multi-author teams with affiliations from different scientific disciplines in orthodontic publications within a decade.[2-3]

The purpose of this text is to review the advances occurring in the field of research methods of investigating various properties of orthodontic applications of materials, to assist the reader in navigating into this topic; a synopsis of the future of materials is also provided listing future applications, which already exist at experimental stage or are yet unavailable but with the relevant technology already presented in broader scientific disciplines.

## EVOLUTION OF ORTHODONTIC MATERIALS RESEARCH APPROACHES: A LIST OF PARADIGMS

The next paragraphs describe briefly the shift, which has occurred within the past decades in the approaches to resolve several issues related to orthodontic materials and their applications. The subheadings lead the reader from the first steps of intervention beginning with bonding and covering the appliances and their properties. This section is complemented with an overview of *in vivo*-ageing studies or retrieval analyses, which constitute the biggest breakthrough in the area of simulation of clinical environment.

### a) Bonding

#### *1. Roughness of enamel*

The alterations in the composition, topography and roughness of enamel has been the topic of research owing to the interest on potential irreversible changes occurring assigned to orthodontic bonding; one of the factors assessed is roughness. The traditional approach has been to employ images of enamel (initially microscopic images and later scanning electron images) to quasi-quantify the effect of bonding on enamel appearance, with apparent lack of sensitivity since only approximations can be made on the roughness variation of surfaces. Later on, a stylus-type profilometric analysis of the surface of the substrate before and after bonding was used to investigate the increase in various roughness parameters, which are used to describe the variation of the surface (presence of peaks, valleys etc). As shown in Figure 1, the conventional approach with the profilometry suffers from various limitations related mostly to the dimensions of the recording stylus; in the case of deep, narrow valleys, with dimensions smaller than the order of magnitude of the stylus, this method does not provide an accurate account of the extent of alterations (Figure 1a). The use of 3D optical profilometry, bypasses this obstacle (Figure 2b), as does the application of Atomic Force Microscopy (Figure 1c), which however is essential for studying surface roughness at the nanoscale level, having resolution far exceeding that of other stylus and optical based methods, and which is somehow not applicable in large surface variations as those expected to occur following bonding.[4-5] In the past 2 decades we have come to understand far more on the topic than we have ever had and this is largely due to the involvement of resources, material and human, from various scientific disciplines.

2. *Polymerization efficiency (degree of C=C conversion)*

The polymerization efficiency also termed degree of cure (DC) or conversion, is often mistakenly noted as ‘degree of polymerization’; the latter represents the number of repetitions of the molecule of monomer to become polymer. The DC is considered a key property for all polymers. In dental composite resins, this variable has been found to modulate the physical, mechanical and biological properties of the material since a poor polymer network is susceptible to release of biologically reactive substances (monomer, additives), predisposes to water absorption and swelling and hydrolytic degradation, and is associated with reduced mechanical properties.[6-9] The latter had given rise to approaches, which focused on the investigation of the phenomenon through mechanical testing with the hypothesis that since, higher degree of conversion is associated with improved mechanical properties profile of the material, evidence on the polymerization efficiency of the polymer could be extrapolated by measuring the response to various tests. However, the kinetics of degree of cure and mechanical properties at the time were not explored, and this resulted in erroneously assigning higher degree of cure in materials which presented better mechanical properties. Since the 1970's though the pioneering work of I. Ruyter,[10] the degree of cure of orthodontic (and broader dental) resinous adhesives has been investigated with the actual measuring of the extent (%) of unreacted methacrylate groups, thereby allowing the percentage of converted double bonds (polymerization) to be accurately estimated on the surface of materials.[11-12]

*3. Bond strength*

Bond strength tests constitute a large portion of bonding-related literature with more than 700 publications listed in the pubmed database during the past decade. Aspects of test standardization related to direction of loads, morphologic variation of teeth crown, source, age, and maintenance of teeth in solutions, [13] have been analyzed and the shortcomings of this preliminary test have been demonstrated.[14] The lack of ageing of materials[15] and the associated questionable clinical relevance of this test has shifted the interest to more meaningful research, which provide an appraisal of the performance of the material complex (adhesive-bracket) in conditions identical to the actual clinical situation. Therefore, failure rate studies adopting a prospective study protocol[16] or even better, an RCT design,[17] have gradually appearing more often in the literature because of the direct clinical extrapolation. Nonetheless, the generalization of results in this case, albeit much wider than its bond strength counterparts, depends on the operator and thus, the results should not be interpreted in their absolute meaning without caution.[18]

*4. Prevention of demineralization*

The attachment of orthodontic appliances to the enamel surface and their prolonged presence in the oral environment has been associated with the development of some unfovarable sequelae in the form of demineralization of the hard dental tissue. Fluoride is the most potent cariostatic agent available that can prevent lesions to develop and dental hygiene plans have this as a central concept with the use of many materials at different stages such as bonding (glass-ionomer cements), and during treatment (gels, rinsing solutions and varnishes). For some of these applications such as the glass-ionomer cements or fluoride-releasing resinous adhesives, the long-term cariostatic effect of fluoride releasing adhesives has not been sufficiently established, since most of the fluoride is released within the first few days or weeks.[19-21] Therefore, topical fluoride in the form of solutions, varnishes or gels have been an integral part of hygiene. A revolutionary recent approach targets the ttreatment of enamel following the formation of white spots; which until previously was limited to interventional restorations. Specifically, peptides such as a statherin-like peptide have been found to reduce the rate of HA demineralization in caries-simulating solutions by about 50%; research efforts also focus on salivary proteins, which can bind to HA surfaces and form a selectively permeable pellicle.[22]

**b)** ***Appliance***

*1. Corrosion*

Corrosion of the orthodontic bracket-archwire complex has received attention after the corrosion products of the bracket base were shown to be diffused into the adhesive.[23] The complexity of the materials and interfaces involved contribute to the development of corrosion in various elements of the appliance. For example, the bracket in its traditional configuration, is composed of two phases: a low modulus of elasticity stainless steel alloy for the manufacturing of the base, which presumably allows for easy debonding after the completion of treatment; and a high modulus steel alloy for the wings, which intends to minimize deformation arising from the engagement of the wire and transfer the stresses from the activated archwire or the prescribed bracket slot, to the tooth.  These two alloys are joined with the use of brazing alloys composed of Ni, Ag or Au and as a result there is a galvanic corrosion formed. In addition, the engagement of the wire into the slot with the use of stainless steel ligatures formulates an environment where many forms of corrosion can be developed. For further information, the reader is referred to a thorough yet simplified review on the topic, which lists all potential forms of corrosion and their mechanism.[24]

The advances on the field of orthodontic corrosion are largely due to the work of engineers, who laid the fundamentals of the understanding of the mechanisms of the phenomenon . The initial methods of plain examination of the surface under incident light (low magnification microscopy) or by weighing the material before and after its exposure to the effector, were substituted by research methods assessing the galvanic potential through measuring the potential differences. Studies on the field identified that the potential differences were found to be positive, indicating that the archwires were consistently the cathode and the brackets were the anode of the galvanic cell. Concurrently, imaging techniques have been advanced and scanning electron microscopy in conjunction with energy dispersive analysis have contributed information on the topography, morphology and elemental analysis of the areas of interest of the alloys.[25]

2. *Forces and moments of wire-bracket combinations in various setups*

The traditional approach of the majority of articles published in the early stages of research development of the field of orthodontic materials consisted of bench tests with simplified laboratory mechanical configurations, which in the majority of cases, were limited to 3-point bending and cantilever tests; tensile tests for the extrapolation of the modulus of the material were also often employed. An effort to simulate the orthodontic clinical analogue had to overcome 2 basic obstacles: the complex mechanical profile of multiple brackets bonded to teeth in an arch form configuration, with wires engaged into the slots and ligated with elastomeric or stainless steel ligatures; and the ageing of the materials in the intraoral environment and resultant effects on the properties of the materials. Whereas the latter was not resolved until recently, the first difficulty was overcome by utilizing a series of approaches:

i) Theoretical analysis with the use of finite element analysis software was introduced in orthodontics from related sciences as a valid tool to predict the forces and moments developed during engagement of in cases of insertion of miniscrews and orthodontic implants.[26]

ii) Furthermore, experimental instrumentation included advanced analytical tools such as X-ray diffraction[27] and Differential scanning calorimetry,[28] which although did not provide a direct result on the actual mechanical profile of the archwires, assisted essentially in explaining the performance of materials in mechanical tests and improved our understanding of the mechanisms underlying the load-deformation curve of these wires.

iii) Concurrently, new complex mechanical testing configurations were proposed to simulate a bonded arch with various clinical malocclusion scenarios and selection of a broad range of bracket and wire types.[29-30] These *in vitro* constructed analogues of the clinical situation provided information, which may be considered as the closest to the intraoral situation if the effect of ageing of the materials is excluded; that was a decisive step in obtaining data on the development of forces and moments at the 3 planes of space during engagement of wires in brackets.

iv) Finally, more recently, brackets with integrated strain gauge-type sensors, were proposed; these are the most advanced appliances from the onset of specialty.[31] These are capable of providing actual force ranges either in real time (in the case of bulky brackets, which carry a power source) or in a retrospective fashion, which allows the manufacturing of slim forms of appliances.

3. *Effect of intraoral conditions on materials*

This area has undergone extensive change, to the point that it constitutes a new field of research activity. Initially, the materials were subjected to ageing by immersing them into various liquids (water, saline, artificial saliva, acidic liquids) for various times and then performing tests (mechanical properties, structural investigations, compositional alterations) to identify the effects induced by ageing. Modern research relies on retrieval analysis, which was already a routine approach in the biomedical literature in the 1970's. The analysis of retrieval analysis goes beyond the scope of this article and the interested reader is referred to an article and a book in this topic.[15]

4. *Effectiveness of materials and techniques*

After the 1990's there has been a specific interest on the investigation of the material performance through clinical trials-as opposed to extrapolation of information on their intraoral behavior by in vitro tests or simulations. This approach has provided information, which rejected assumption on various topics such as self-ligating brackets, effectiveness of NiTi wires, or failure rate of brackets; further information on this topic can be found in this journal issue.

***c) Side effects of materials***

Within this field, the release of substances (ions from alloys, and monomers, degradation byproducts and additives from polymers), and the biological properties of materials are included.

1. *Release of substances from orthodontic materials*

At the early stages, efforts consisted of measuring *in vitro* and with primitive techniques (weight, morphology), the ions or monomers released in immersion media. Later, the same method was complemented with the introduction of instrumental analysis such as atomic emission or absorption spectroscopy[32] for the case of metals; high performance liquid chromatography and gas chromatography-mass spectroscopy were also employed for the qualitative and quantitative analysis of immersion media, saliva, blood or urine with respect to concentration of polymer byproducts.[33-37]

2. *Biological properties of materials*

With the establishment of a reliable method for the identification of released substances, the necessity for assessing the potential biological reactivity emerged. The earliest methods in the field were usually confined to adding various concentrations of effectors hypothesized to be released from orthodontic materials in cultures of immortal cell lines and observing the inhibition zone, which correspond to the lack of level of concentration causing cell death. Later more advanced techniques allowed for the assessment of action at subtoxic concentrations to observe the effect on the physiology of cell (or animal) by using markers of oxidative stress or metabolic function and in 3D cultures.[38-40] Then, with the availability of data on the quantity of species released, these levels of effectors can be added to human gingival or periodontal ligament fibroblasts to secure two things: first the corresponding mode of action at doses documented to occur in vivo and secondly to identify potential effects on human cells.

## THE FUTURE OF ORTHODONTIC MATERIALS

1. *Shape memory polymers*

Shape-memory polymers are an emerging class of polymers with applications spanning various areas of everyday life. These polymers are dual-shape materials belonging to the group of “actively moving” polymers that can change from one shape to another, the first being a temporary shape obtained by mechanical deformation and the second obtained from subsequent fixation of that deformation.[41]

For shape-memory polymers, heat, light, infrared radiation, electrical and magnetic fields, and immersion in water have been used to induce this property. The shape-memory effect depends on the molecular architecture and does not require a specific chemical structure in the repeating units. In essence, application of external stimuli to these materials introduces a departure from the as-received shape to a new configuration that is reversible.

In orthodontics, the potential application of these materials involves the manufacturing of polymeric transparent wires with minimum stiffness, which could then be transformed into archwires of predetermined elastic modulus upon exposure to a stimulus such as light or heat.

Therefore, aesthetics along with a preferable stiffness would be attained during intraoral application of these materials.

2. *Self-healing materials*

The design and manufacturing of “smart” synthetic systems that can mimic the behavior of biological systems which heal themselves has been an objective of intense research during the past decade. Hybrid materials have been recently created, where micron-scale conduits extend throughout the material and contain healing fluids or dissolved healing agents.[42] When a crack appears near the network, the fluid can flow to the damaged region and fill the fissure. The simplest form to achieve this behavior might be the incorporation of bubbles of a material/substance/precursor in the raw material. This reservoir, upon exposure to air, polymerizes as a result of crack formation and spontaneously closes the crack, thus maintaining the structural integrity of the material.

The orthodontic application of this concept may involve polymer brackets and archwires. The integration of nanosized bubbles filled with autopolymerized monomer in these materials may result in reduction of wire and bracket breakages. Fracture of the bracket or wire would induce bursting of the nanobubbles and exposure of the monomer to air, thereby resulting in polymerization and filling of the crack-induced gap.

3. *Biomimetic adhesives*

The issue of an enamel-friendly bonding mechanism for orthodontic appliances has been the subject of investigations since the original introduction of the acid-etching technique. This intense interest derived from the description of alterations of enamel color and structure associated with acid-etched-mediated bonding . Although glass-ionomer adhesives have offered an alternative, their applications remain limited, probably because of the higher failure rates.

The introduction during the past 15 years of a new class of materials that adopt the paradigms of nature has gradually established the category of biomimetic materials. This term derives from Greek “bio” (living) and “mimetic” (imitating or resembling), and refers to how creatures ingenuously employ natural elements to solve problems in the environment.

Geckos, for example, are lizards that belong to the species of *gekkonidae* are characterized by a remarkable ability to sustain their weight while upside down. The strong but temporary adhesion employed by a gecko comes from a mechanical principle known as “contact splitting”. The foot of a gecko has a flat pad which is densely packed with very fine hairs that are split at the ends, resulting in a greater number of contact points than if the hairs were not split. More contact points between these hairs and a surface result in a significant increase in adhesion force. Researchers have discovered that this special nature of the foot pads allows the gecko to stick to surfaces through the formation of localized van der Waals forces.[43] This mechanism has been employed for high-friction microfibers or carbon nanotubes, which are sprayed on a surface. Because of their enormous number per unit area, the physical forces developed mimic the ability of a gecko to attach firmly to surfaces without the use of a chemical substance. While this mode of bonding may be suitable for dry environments, it fails to provide reliable performance for wet surfaces. This problem inspired researchers to adopt another natural example of bonding: that of mussels. Combining the important elements of gecko and mussel adhesion, the new adhesive material, called "geckel", functions like a sticky note and exhibits strong yet reversible adhesion in both air and water.[44] Mussel-mimetic polymers have an amino acid called L-3,4-dihydroxyphenylalanine (DOPA) found in high concentrations in the "glue" proteins of mussels. Analogously to the gecko-based approach, pillar arrays (400-600 nm in diameter and length) coated with the mussel-mimetic polymer improved wet adhesion by 15-fold over uncoated pillar arrays.

The orthodontic application of this innovation is profound. Brackets having bases with pads mimicking the gecko foot and covered with a layer of DOPA would provide adequate bond strength to sound enamel without prior enamel conditioning and with minimal color and structural alterations to the enamel. To the knowledge of the authors, use of this type of biomimetic adhesive has not yet been adapted by a manufacturer to orthodontic brackets.

*4. Self-cleaning materials*

The issue of plaque retention on brackets and microbial attachment onto these calcified biofilms has been a major concern from an enamel prophylactic perspective. The development of a material that could clean itself not only from inorganic mainly, but also from organic, precipitations is an application that is very attractive in the materials science areas involving biomedical, industrial and aeronautical applications.

Early research in this field adopted the paradigm of microscopic bumps on a lotus leaf, which transform its waxy surface into an extremely water repellent, or superhydrophobic, material. Synthetic self-cleaning materials have been developed, some of which are based on this "lotus effect", whereas others employ the opposite property of "superhydrophilicity" as well as catalytic chemical reactions.

The use of titanium oxide ($TiO_x$) nanocoatings has been found to increase the safety of aircraft by preventing the buildup of ice and other contaminants. This process creates self-cleaning "superhydrophobic" surfaces, and involves the application of a hydrofluoric acid coating on the titanium alloy substrate. A substantially self-cleaning superhydrophobic surface is created when exposure to ultraviolet light causes the titanium oxide layer to undergo photocatalytic reaction with oxygen to oxidize any organic contaminants that may be present. Since superhydrophobic surfaces resist soiling by water-borne contaminants, they are easily cleaned and useful in directing flow in microfluidic devices.

Photocatalytic activity from the reaction of titanium oxide with ultraviolet light has recently gained attention in orthodontic materials.[45] In particular, there is scientific interest in inducing photocatalytic reactions on the nickel-titanium archwire alloy. By thickening the titanium oxide film with electrolytic treatment and then applying heat treatment, the surface film on NiTi was modified from an amorphous structure to crystalline rutile ($TiO_2$).

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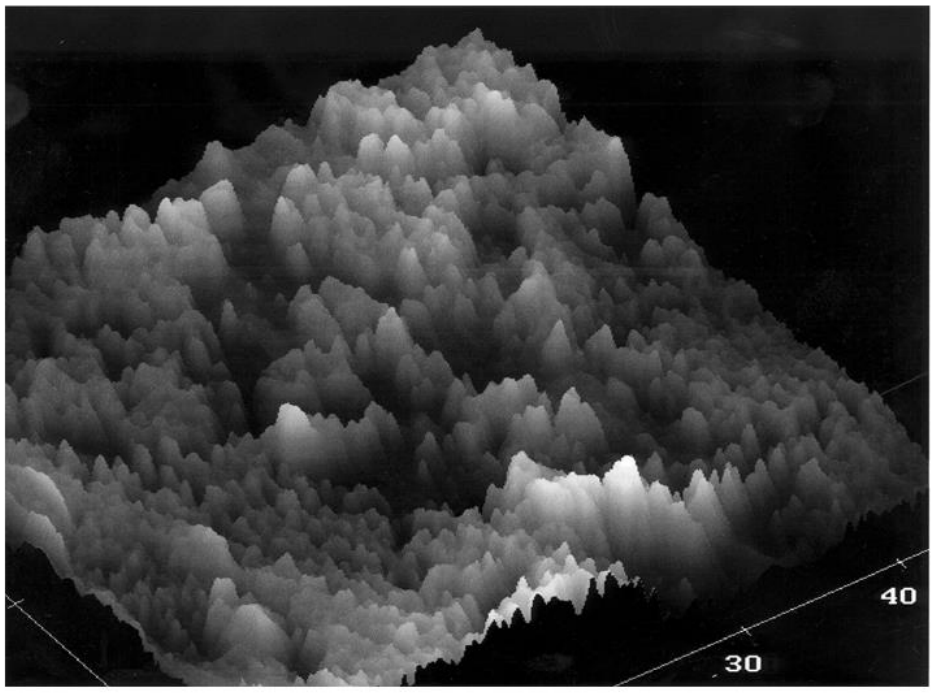
FIGURE LEGENDS

Figure
Various methodologies used to assess roughness.

a) Graph of the profilometry utilizing a stylus method demonstrating the problems in assessing the actual variation of peaks and valleys on a surface establishing the limiting factor of stylus size.
b) Representative 3D optical profilometric images of all groups tested; IE: intact enamel; AE: acid etched enamel (adopted from Patcas et al Am J Orthod Dentofacial Ortho in press
c) Atomic force microscopic image depicting the high resolution (nanoscale range) of the method.



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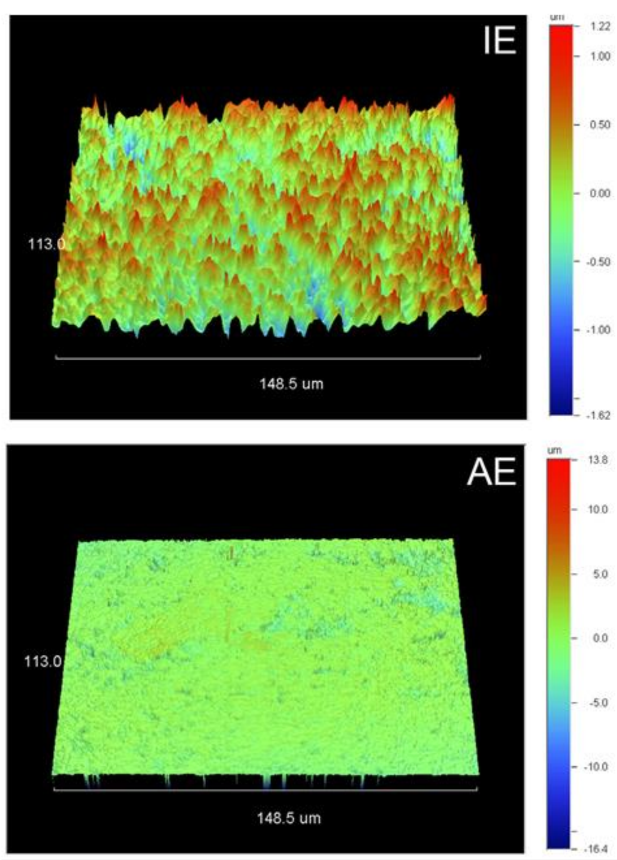

IE
113.0
148.5 um
um
1.22
1.00
0.50
0.00
-0.50
-1.00
-1.62
AE
113.0
148.5 um
um
13.8
10.0
5.0
0.0
-5.0
-10.0
-16.4